



GLASS BEADS FOR PAVEMENT MARKING MATERIAL MSP-99-02D

1.0 Description. These specifications cover Type I free flow, Type I moisture resistant and Type L (Large) glass beads for drop-on application on traffic marking materials for the production of a reflective surface to improve the night visibility. The beads shall be highly resistant to traffic wear and to the effects of weathering.

2.0 Materials. Type I and Type L beads shall meet the requirements of AASHTO M 247, Type I, except as noted herein.

2.1 Coating. Type L beads shall be coated to insure satisfactory embedment and adhesion when applied to an uncured traffic marking material. Type I beads shall not be embedment coated.

2.2 Roundness.

2.2.1 Type I beads shall have a minimum of 70 percent true spheres when tested in accordance with ASTM D 1155.

2.2.2 Type L beads shall have a minimum of 80 percent rounds per screen for the two highest sieve quantities (determined visually) and no more than 3 percent angular particles per screen (visual). The remaining sieve fractions shall typically be no less than 75 percent rounds (determined visually per aspect ratio using microfiche reader). The tests shall be in accordance with Federal Lands Highway (FLH) Test Method T520-93.

2.3 Gradation. Type L beads shall meet the following gradation requirements when tested in accordance with ASTM D 1214.

U. S. Standard Sieve No.	Percent Passing
12 (1.7 mm)	100.00
14 (1.4 mm)	95 - 100
16 (1.18 mm)	80 - 98
18 (1.00 mm)	10 - 42
20 (850 μ m)	0 - 7
25 (710 μ m)	0 - 2

2.4 Silica Content. The beads shall be made of glass containing not less than 58.0 percent Silica (SiO_2) when tested in accordance with ASTM C 169, Procedures for Referee Analysis.

2.5 Water Resistance. The beads shall show no readily discernible dulling and the amount of 0.1 Normal Hydrochloric Acid needed to titrate the filtrate shall not exceed 4.5 ml, when tested in accordance with this specification.

2.6 Calcium Chloride Resistance. The beads shall show no readily discernible dulling when tested in accordance this specification.

2.7 Sodium Sulfide Resistance. The beads shall show no readily discernible darkening or dulling when tested in accordance with this specification.

3.0 Test Methods.

3.1 Water Resistance. Ten \pm 0.5 grams of beads placed in a Whatman single thickness cellulose extraction thimble, 33 by 80 mm, are refluxed for one hour in a Soxhlet extractor having an 85 mm siphon capacity using 150 ml of distilled water. All connections shall be ground glass. At the end of the refluxing period, allow the filtrate to cool to room temperature, and titrate with 0.1 normal hydrochloric acid, using phenolphthalein indicator. The beads shall be dried at 100 C, and examined for dulling under 60-power magnification.

3.2 Calcium Chloride Resistance. Immerse approximately 10 grams of the beads in a 1.0 Molar calcium chloride solution for 3 hours. Rinse well, by decantation, with distilled water. Spread beads on a clean filter paper and allow to dry. Examine the beads for dulling under 60-power magnification.

3.3 Sodium Sulfide Resistance. Immerse approximately 10 grams of the beads in a 50 percent solution of sodium sulfide for one hour. Rinse well, by decantation, with distilled water. Spread beads on a clean filter paper and allow to dry. Examine the beads for dulling under 60-power magnification.

3.4 Embedment Coating. The embedment coating on Type L beads shall be tested in accordance with the following procedure.

3.4.1 Apparatus and Reagents.

Graduate Cylinder 50 ml	Glass Filter Paper 100 mm diameter
Dansyl Chloride - 98 percent	Scale - Analytical Balance (4 place)
Acetone - Reagent Grade	50 mm Buchner Funnel and Suction Flasks
Safety Glasses or Goggles	Darkened Glass Container (that can be sealed tightly)
Rubber Gloves (long sleeves)	Small Aluminum Weighing Dishes
Medicine Dropper	50 mm diameter Filter Paper (Whatman #1)
Vacuum Pump	Ultra-Violet Light Source - Intensity 7,000 uw/cm ²

Caution: Dansyl Chloride is a hazardous compound. Do not handle without protective gloves and safety glasses or goggles. Do not get onto skin.

3.4.2 Preparation of Dansyl Chloride Solution. Prepare a solution by weighing 0.2 grams of Dansyl Chloride and dissolving it in 25 ml of acetone. This solution can be used for several tests during the day but must be kept refrigerated in a dark, tightly closed container between uses. Make a fresh solution daily.

3.4.3 Procedure.

- (a) Set drying oven to 60 C. Turn on ultra-violet light.
- (b) Weigh 2 samples of beads of 10 grams each. Place the sample to be evaluated in an aluminum weighing dish. Retain the other sample for a fluorescence observation comparison.
- (c) Place a 50 mm diameter filter paper into the Buchner funnel and attach to the suction flask.
- (d) Put the beads in the Buchner funnel and saturate the sample with the Dansyl Chloride solution using a medicine dropper. Let solution and sample stand for 30 seconds.
- (e) Place the saturated beads into an aluminum dish and dry in an oven at 60 C for 15 - 20 minutes. Beads will be yellow and agglomerated. Do not let the Dansyl Chloride solution char. (Properly discard the used filter paper because of the toxicity of the Dansyl Chloride.)

- (f) Remove sample from the oven and place the glass beads in the Buchner funnel with new filter paper. Rinse the beads with 100 ml of Acetone. Use the suction during this step. All yellow must be removed from the beads.
- (g) Remove the beads from the funnel and place into a new aluminum tray. Allow the beads to dry in the oven for 5 - 10 minutes until free flowing.
- (h) Remove the beads from the oven and place on glass filter paper. If beads are agglomerated, break them up with a spatula.
- (i) Inspect the treated sample under the ultra-violet light, in a darkened room.

3.4.4 Observations.

- (a) Embedment coated beads will emit a yellow-green florescence.
- (b) If additional fluorescence is observed when compared with the original untreated sample, the lot is accepted. If no additional fluorescence is observed, the test should be rerun using a new 10 gram sample of beads and a fresh solution of Dansyl Chloride.
- (c) If no additional fluorescence is observed on the new sample of beads, the material is not properly coated and the lot is rejected. If additional fluorescence is observed, the lot is accepted.

4.0 Sampling and Testing. The manufacturer shall furnish the engineer free access to all parts of the plant and shall furnish every reasonable facility for inspection.

4.1 The engineer reserves the right to sample at the point of manufacture, at intermediate points of storage, or at destination. The engineer will determine the location and frequency of sampling.

5.0 Certification and Acceptance. The contractor shall furnish a manufacturer's certification to the engineer for each lot, certifying that the beads conform to all requirements of these specifications. The certification shall include or have attached specific results of tests performed for roundness, refractive index, flow properties, coating and gradation. The certifications shall also show the quantity and lot number.

5.1 Pre-approved beads may be accepted by certification and random sampling as designated by the engineer. Beads which have not been pre-approved must be sampled and approved prior to use.